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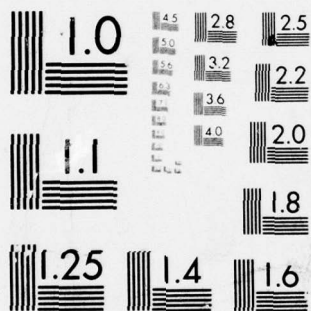
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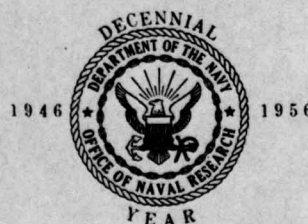
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symposium
technology of
MOLYBDENUM
and its alloys

ONR Symposium Report
ACR-12

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Rackham Memorial Building
Engineering Society of Detroit
September 18-19, 1956



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Chairman: O. T. Marzke
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SESSION I--September 18, 1956, at 9:45 a.m.

MOLYBDENUM AS A STRUCTURAL MATERIAL

Chairman: H. B. Goodwin
Battelle Memorial Institute

**PROPERTIES AND APPLICATIONS OF COMMERCIAL
MOLYBDENUM AND MOLYBDENUM ALLOYS**

R. R. Freeman
Climax Molybdenum Company

One of the distinctive advantages of molybdenum over other ultra-high-temperature materials, such as cermets and many superalloys, is the commercial availability of both the alloyed and unalloyed molybdenum in a wide variety of forms, including plates, sheets, bars, wire, and tubing.

Molybdenum is readily machinable, and can be fabricated by the usual metal-working processes of forging, rolling, drawing, and spinning. Satisfactory welding, brazing, and riveting procedures have been developed.

Of all the many outstanding properties of molybdenum, high strength at elevated temperatures, high melting point, and excellent thermal and electrical properties are of primary importance for most applications. Molybdenum now finds wide use in industries as diverse as glass, electronics, machine tools, missiles, and atomic energy. Molybdenum alloys have been developed which retain useful strength properties up to at least 2000°F, and it is anticipated that this development will speed the application of molybdenum to many new fields.

SESSION II--September 18, 1956, at 11:30 a.m.

**PREPARATION AND FABRICATION OF
MOLYBDENUM AND ITS ALLOYS**

Chairman: L. F. Yntema
Fansteel Metallurgical Corporation

CONSOLIDATION OF MOLYBDENUM BY POWDER METALLURGY PRACTICE

H. Scott and D. D. Lawther:
Chief Metallurgist
Westinghouse Research Laboratories

and

W. A. Taebel
Superintendent
Special Molybdenum Products Department, Westinghouse Lamp Division

Molybdenum, like most other metals, is extremely sensitive to atmospheric contaminants. These contaminants are readily introduced into metal powders by exposure to air, and have major detrimental effects on the mechanical properties of the consolidated metal. Since molybdenum goes through the powder stage during refinement, elimination of such contamination is a basic problem in the consolidation of molybdenum by any means. The oxidation characteristics of molybdenum are reviewed in the first section of this paper for their bearing on the art of producing molybdenum metal and its alloys by powder metallurgy techniques.

Current practices for production of wrought molybdenum from sintered powders are outlined in the second section. Major difficulties mentioned with the control of final mechanical properties are mentioned, along with the steps taken for their elimination. The importance of the transition from ductile to brittle fracture under tension at atmospheric temperatures is emphasized, as well as the need for eliminating grain boundary fracture.

Alloying with reactive metals for increased strength at room and elevated temperatures is then considered, with particular emphasis on the problem of lowering the transition temperature at the same time. This work, conducted in the laboratory only, employed vacuum sintering exclusively for the attainment of low oxygen compacts, which was found to be essential for successful hot working. Substantially higher alloy contents were workable when sintered than when produced by arc casting. Surprisingly large increases in the recrystallization temperature of molybdenum were found, particularly with the use of zirconium as an alloying element.

The alloys were tested for creep-rupture strength at 1800°F, and showed marked improvement over non-alloyed molybdenum. Tests were made in a vacuum, with results which contrasted sharply with those of other investigators. The inconsistency appears to result from differences in the degree of vacuum used and, though incompletely resolved, points clearly to further possibilities for improvement of creep-rupture strength.

ARC-MELTING MOLYBDENUM

R. G. Yingling and G. A. Timmons
Climax Molybdenum Company

Arc-cast molybdenum is now being produced regularly in special equipment in which molybdenum powder is automatically compacted into an electrode which is sintered to increase its strength, and then melted by an ac arc in a water-cooled, copper crucible under vacuum at 20-40 microns. The raw material is hydrogen-reduced powder of approximately 4-7 microns in size, and the product of arc casting is ingots up to 9-in. diameter weighing 1,000 pounds. The pressing, sintering, and melting of the consumable electrode occur simultaneously within the same enclosure.

To be satisfactory for forging and other hot working processes, arc-cast molybdenum must contain less than 0.005 percent of oxygen. Since hydrogen-reduced molybdenum powder usually contains 0.015-0.04 percent of oxygen, some carbon must be added to the charge to effect the required deoxidation when melting is accomplished in a vacuum. The process makes use of the turnings which result from the scalping of ingots and extrusions. Alloying elements are added to the powder charge in small particles of the purest form now commercially available.

Although most of the arc-cast molybdenum supplied to industry today is melted in vacuum, it has been demonstrated that forgeable molybdenum may be produced by melting under argon or argon-helium atmospheres, using aluminum as the deoxidizer.

At the present stage of development, operations are conducted in the fourth PSM machine; this machine was built in 1950. Arc power is provided by a single-phase, air-cooled transformer rated at 360 kva. The melting rate is approximately 10 pounds per minute, and power consumption is of the order of 0.5 kwh per pound. A crew of three men is employed to operate the machine.

Accessory equipment required for the arc-casting operation comprise a V-blender for mixing the charge, overhead crane for raising the loaded hopper to its position atop the main chamber, hand-operated elevator truck for handling molds and ingots, hack saws for cropping and sectioning the ingots, lathes for turning the ingots to remove surface defects and to provide a standard size for extrusion, and a roller crusher for reducing and flattening lathe chips to the size and shape suitable for incorporating in the powder charge. Each ingot is inspected by examining a fracture of a section cut from the midlength position; the ingot is also analyzed for carbon and the added alloying element.

Some molybdenum ingots have been produced experimentally by melting pellets of molybdenum powder in vacuum and in inert atmospheres with the dc arc, using water-cooled, tungsten-tipped electrodes, and some experiments have been performed in which prefabricated molybdenum electrodes have been melted with dc arc under vacuum and under inert atmospheres.

WORKING OF MOLYBDENUM AND ITS ALLOYS

J. D. Nisbet and W. L. Bruckart
Universal-Cyclops Steel Corporation

The basic theory of the hot working of metals having widely different melting points is reviewed. These principles are applied to molybdenum and molybdenum-base alloys, and the temperature ranges for hot and cold working are developed. It is pointed out that all present methods for fabricating molybdenum are used near the recrystallization temperatures, therefore working under present conditions is achieved in an intermediate range between truly hot and truly cold working temperatures. Present methods and techniques for fabricating molybdenum and molybdenum-base alloys are reported. Several laboratory examples designed to achieve temperatures and atmospheric conditions for truly hot working of molybdenum are cited and reviewed.

A large pilot plant designed for the fabrication of molybdenum and molybdenum-base alloys at very high temperatures and in an inert atmosphere is described. Some of the engineering problems confronted in designing such a pilot plant are reviewed.

JOINING OF MOLYBDENUM AND ITS ALLOYS

W. N. Platte

Westinghouse Electric Corporation

The influence of the contaminating elements, oxygen and nitrogen, on the soundness and mechanical properties of molybdenum fusion welds is discussed. Deoxidization of the base material is shown as a means of producing sound welds and reducing the brittle-to-ductile transition temperature. The damaging effects of oxygen and nitrogen on the soundness and mechanical properties of molybdenum welds are shown. The interactions between oxygen and nitrogen when used in combination in argon welding atmospheres are shown with respect to their effects on the weld metal properties. Welding limits are suggested for successful welding of molybdenum.

A discussion is given on the improvement of properties of molybdenum-welded joints by the use of materials with specific deoxidizer and alloy additions. The use of alloys of molybdenum is shown to be the most effective way to improve the ductility at low temperatures. Welds have been obtained which bend 118° at room temperature by the use of high-purity welding atmospheres and proper alloy additions.

DINNER SESSION--September 18, 1956, at 8:00 p.m.

EUROPEAN DEVELOPMENTS ON MOLYBDENUM

Chairman: O. T. Marzke
Naval Research Laboratory

MOLYBDENUM RESEARCH AND DEVELOPMENT IN GREAT BRITAIN

L. Northcott
Materials Division
Armament Research and Development Establishment
Woolwich, England

An outline is given of some investigations now proceeding in England upon metallic molybdenum, together with views as to fields requiring further study.

Active work is giving a better understanding of the arc melting process by a study of the physics of the arc. Refinement of crystal structure is being attempted by rotation of the liquid metal during solidification. In view of the detrimental effect of interstitial impurities, such as oxygen, carbon, and nitrogen, purification processes are being studied, including zone melting. Since molybdenum has a body-centered-cubic structure, the general properties of metals with such a structure are reviewed, and the possibilities of alloying to give a face-centered-cubic structure are considered. Work is proceeding on the hot working characteristics of molybdenum both in air and in a controlled atmosphere, the present indications being that the energy required for deformation is much less and the malleability of the metal much greater at temperatures approaching 1800°C than at lower temperatures. Work is proceeding on the constitutional aspects of molybdenum-rich alloys from the standpoint of alloy theory. Methods of applying coatings of molybdenum by electro-deposition, by vapour deposition, and by spraying are under examination, and further work is in hand on the preparation of oxidation-resistant coatings on molybdenum.

SESSION III--September 19, 1956, at 9:00 a.m.

METALLURGY OF MOLYBDENUM AND MOLYBDENUM-BASE ALLOYS

Chairman: J. B. Johnson
Aeronautical Research Laboratory
Wright Air Development Center

THE DUCTILE-TO-BRITTLE TRANSITION IN MOLYBDENUM

J. H. Bechtold
Research Laboratories
Westinghouse Electric Corporation

Molybdenum, like other metals with the body-centered-cubic crystal structure, undergoes an abrupt transition from ductile-to-brittle behavior. This transition in ductility appears to occur near room temperature when molybdenum is tested by the conventional static tension test. The temperature of the transition depends, however, upon the method of testing, and will occur at much higher temperatures in more severe tests, such as the notched impact test, or at much lower temperatures in a torsion or compression test.

The transition phenomenon in molybdenum is very similar to that observed in other body-centered-cubic metals. The main difference between the mechanical properties of molybdenum and that of other body-centered-cubic metals is in the temperature range over which the transition occurs. However, this temperature range is that which is expected, based upon the modulus of elasticity of molybdenum.

The basic reason for a transition temperature is the rapid increase in the yield strength with decreasing temperature. This increase occurs over a characteristic temperature range for different body-centered-cubic metals, and is the factor which determines the temperature range in which we can expect to find the transition in ductility. Brittle fracture, without prior microscopic yielding, occurs when the yield strength exceeds the stress at which a crack will nucleate and propagate. This stress is well below the theoretical cohesion strength of the metal. The temperature dependence of the ductility above the temperature of complete brittleness is determined in part by the relative effects of strain on the yield strength and the fracture strength. Although the yield strength is the factor of greatest fundamental importance, and the one which determines the temperature range in which the transition in ductility will occur, the fracture characteristics are the factors which are most strongly affected by chemistry and microstructure and which are, in general, responsible for the variations in transition temperatures observed from sample to sample.

The effects of mechanical, metallurgical, and chemical variables on the transition temperature are discussed with respect to their effect on the basic flow and fracture characteristics of molybdenum.

THE INFLUENCE OF CARBON, OXYGEN, AND NITROGEN ON THE DUCTILITY OF MOLYBDENUM

J. Wulff

Department of Metallurgy
Massachusetts Institute of Technology

Tensile test data, obtained at temperatures from -193°C to $+100^{\circ}\text{C}$ on molybdenum refined in nine passes by a floating zone technique, indicate that at liquid nitrogen temperatures the material still exhibits a 12-percent reduction in area. Commercial molybdenum wires of 0.010-inch diameter and of various carbon and oxygen contents were similarly tested in (1) the stress relieved, unrecrystallized state, (2) after a 30-minute heat treatment at 2100°C in mercury diffusion pump vacua followed by quenching and (3) slow cooling from 2100°C . Plotted data suggest an apparent dependence on grain size and a real dependence on form and distribution of carbon and presumably also oxygen. Further experiments on wires initially 0.01 wt. percent carbon and 0.006 wt. percent oxygen, treated for 30 minutes at 2100°C in a mercury diffusion pump vacuum followed by half-hour treatments at lower temperature, illustrate the embrittling effect of carbide precipitation. Similar experiments with decarburized wires illustrate the solution of oxides at 2100°C and their reprecipitation at lower temperatures. Precipitation of nitrides could only be followed by heat-treating in an atmosphere of nitrogen. All of the gross precipitation data can be explained with the aid of three tentative solubility curves. These are based on an extrapolation of published metallographic, internal friction, and lattice parameter studies.

From auxiliary experiments, using a mass spectrometer in series with an all-glass vacuum system, it was found that removal of C, O, and N from molybdenum wire below about 1600°C occurs by the emission of CO, CO_2 , and N_2 . This undoubtedly also occurs above 1600°C , but such gases are then "gettered" by molybdenum vapor. In the absence of carbon, oxygen probably leaves the surface of the wire as MoO_3 , and not as atomic or molecular oxygen. Evidence can be shown for the existence of similar refining reactions in arc casting and in zone refining.

DEVELOPMENT AND PROPERTIES OF ARC-CAST MOLYBDENUM ALLOYS

M. Semchyshen
Climax Molybdenum Company

High melting point, availability, and ease of production by the arc-cast process make molybdenum a logical choice for high-temperature structural applications. While pure molybdenum is satisfactory for a number of these, the increased strength attainable through alloying widens the field of use considerably.

In the development of molybdenum-base alloys, some twenty alloying elements, Al, Be, B, Ca, C, Cr, Co, Fe, Mg, Mn, Ni, Nb, N, Si, Ta, Ti, U, V, W, and Zr, were studied. The alloy additions were made to the extent of appearance of the first indication of a second phase. Of the alloys containing two-phase microstructures, only the molybdenum-beryllium alloys responded to precipitation hardening.

The hardness data obtained over the temperature range from 75°F to 3000°F on small heats of binary and ternary molybdenum-base alloys served as the basis for selection of the alloying elements and compositional ranges used for large heats. The hardness data reflected solid-solution hardening effects, because increasing the alloy content increased the hardness over the entire temperature range of hardness measurements.

Since the underlying objective of the investigation was to develop materials for service at elevated temperatures, the obvious alloy compositions for extensive study would be those having the highest hardness at elevated temperatures. Nevertheless, because of inability to fabricate large sections of molybdenum-base alloys of the highest hardness at the temperatures and with the equipment available, it was necessary to compromise on alloy content in the interest of fabrication.

All of the alloys studied were of the solid-solution type, so an additional increase in strength of the alloys was brought about by the mechanism of strain hardening. To evaluate the usefulness of strain-hardened structures for elevated temperature service, recrystallization temperatures were determined, and subsequent studies of mechanical properties were made in both the strain-hardened and the fully recrystallized conditions.

Among the more important mechanical properties determined were hardness from 75°F to 3000°F, tensile properties at 75°F and 1600°F, and creep-rupture properties at 1600°F to 2000°F. As a result of these studies, alloys were found which demonstrated short-time tensile strength at 1600°F of 100,000 psi. The strongest of these alloys exhibited 100-hour creep-rupture life at 70,000 psi at 1800°F, and at 50,000 psi at 2000°F. This represents greater strength than that of any other metallic material known.

POWDER-METALLURGY MOLYBDENUM-BASE ALLOYS

R. I. Jaffee
Battelle Memorial Institute

Molybdenum-base alloys prepared by powder-metallurgy methods have an undeniable advantage over arc-cast alloys in their hot-breakdown fabrication characteristics. Powder-metallurgy alloys typically have fine grain sizes as sintered, and may be breakdown-fabricated by rolling or forging without the preliminary extrusion operation essential for the arc-cast materials. Strength properties of powder-metallurgy molybdenum alloys are potentially as high as those of arc-cast alloys, if advantage is taken of oxide dispersions. For metallic alloying additions, the powder-metallurgy alloys appear inferior. Secondary fabrication of powder-metallurgy alloys is about the same as that of arc-cast material. However, because of inherent higher oxygen contents powder-metallurgy alloys have much poorer fusion weldability.

Molybdenum powder usually has about 0.2-0.5 percent oxygen present as a surface film; even freshly reduced molybdenum powder contained in sealed cans under nitrogen has 0.02-0.04 percent oxygen. This fact dominates the methods used for consolidation and the potential alloying additions. The most suitable atmosphere for sintering is hydrogen, because the surface oxygen may be readily reduced to less than 0.01 percent during sintering. The metallic alloying additions must be limited to those not highly reactive with oxygen, in order to avoid high residual oxygen contents and resultant poor fabrication characteristics. The reactive metals may be added in the form of stable oxides. In this case, hydrogen sintering results in satisfactory densification and a readily workable material. Inert-atmosphere sintering with argon is possible, but this requires the reduction of oxygen by dissociation of molybdenum oxide. Exceptionally pure argon, which is difficult to achieve in practical sintering equipment, is required for this. It would be feasible to reduce the surface films of oxygen with carbon during the sintering process in argon atmospheres, but little work has been done along this line. Vacuum sintering is a possible method for consolidation. However, high sintering temperatures are required, and excessive loss by volatilization results. Vacuum-sintered molybdenum alloys have much lower oxygen contents, but in terms of mechanical properties and fabricability, appear to have no advantage over hydrogen-sintered alloys.

The traditional method of sintering molybdenum by resistance in so-called "hydrogen bottles" works very well for molybdenum alloys. There is no limitation on the sintering temperature, and time-temperature programming is readily done because of the good control possible with this method. The major limitation of bottle sintering is the size of bar that can be sintered, a maximum of about 2 square inches. Furnace-heating methods have been worked out successfully by the Westinghouse Electric Corporation. In this method, lower sintering temperatures are made possible through the use of longer times, such as 8-16 hours at 1750°C. Large-size bars up to 6-8 square inches and a length of several feet can be sintered by this method. Since the entire bar is heated to the sintering temperature, there are no end losses, such as occur with resistance-bottle sintering.

In the final fabrication of molybdenum alloys, a compromise must be achieved between the high ductility and the lower recrystallization temperature associated with high percentages of reduction. Generally, the best compromise is achieved with final percentages of reduction after recrystallization of 60-75 percent in thickness. Like most molybdenum alloys, powder-metallurgy alloys become brittle when recrystallized at elevated temperatures. However, if the grain size is kept fine, there is a range of conditions in which molybdenum alloys will exhibit good tensile ductility when recrystallized. However, because of the higher oxygen content characteristic of powder-metallurgy molybdenum, the ductility of the recrystallized powder-metallurgy alloys probably is somewhat poorer than that of arc-cast alloys similarly treated.

Powder-metallurgy alloys may be made with conventional alloying additions. The traditional molybdenum alloys used extensively in the electronics and lamp industries are molybdenum-tungsten alloys with 20-50 percent tungsten. These alloys have good fabrication characteristics, are handled by the same methods as used for unalloyed molybdenum, have improved recrystallization characteristics, and have improved strength. However, tungsten has the lowest specific strengthening effect of any alloying addition and, because of high density, is not as advantageous from a strength-weight point of view. The most effective metallic alloying addition is silicon; significant improvements in strength at room and elevated temperatures may be achieved with alloy contents on the order of 0.1-0.2 percent silicon. However, silicon losses in sintering tend to be excessive, and control of the element is difficult. Chromium, a good alloying addition, may be added up to 1/2 percent, resulting in improved room and elevated-temperature strength and increased recrystallization temperatures. Cobalt has a high strengthening effect, but it lowers the recrystallization temperature, and is not suitable for elevated-temperature applications. Nickel is probably the worst alloying addition, because it is very embrittling and lowers the recrystallization temperature. This is unfortunate because of nickel's beneficial effect on oxidation resistance. It is possible to secure better over-all strength-ductility characteristics with ternary alloys than with binary alloys, although the specific strengthening effects with ternary alloys appear to be somewhat lower. The reason for this is that the fabricability range is considerably increased in suitable ternary alloy combinations.

High residual oxygen is detrimental in conventional molybdenum alloys because of excessive molybdenum oxide at the grain boundaries. However, stable oxide additions to molybdenum result in effective dispersion-hardening characteristics. The most suitable dispersion oxides for molybdenum are those which have high hardness (to avoid stringing during fabrication) and high modulus of elasticity. The oxide powders should have exceptionally fine particle size, preferably 1 micron or less. They should be sound, so that they do not crumble during deformation. They need high stability with respect to decomposition, reduction by hydrogen, and melting. Finally, a good bond must be achieved between the oxide dispersion and the metallic molybdenum matrix. An experimental survey of potential dispersion agents in molybdenum resulted in the finding that zirconium dioxide and titanium dioxide were most effective. Chromium oxide is good, but somewhat less effective.

Dispersions characteristically have little effect on the low-temperature behavior of materials, and they improve high-temperature behavior. Alternatively, this might be stated that the temperature range at which low-temperature behavior changes to high-temperature behavior is increased by dispersions. Thus, the room-temperature strength and ductility are not changed significantly by the oxide dispersions, but the high-temperature rupture strengths of molybdenum are tripled or quadrupled. The slope of the stress-rupture curve is reduced markedly, and the mode of fracture changes from intergranular cracking to shear failure.

The oxide dispersion alloys of molybdenum with 0.1 to 1 percent of ZrO_2 or TiO_2 were found to have 100-hour rupture strengths of 30-40,000 psi, at 1800°F compared with 10-15,000 psi for unalloyed molybdenum. Recrystallization characteristics are also improved. The dispersion-hardening effects are inherent, and are not dependent on the presence of hot-cold work. Thus, oxide-dispersion molybdenum alloys in the recrystallized condition have improved properties compared with recrystallized molybdenum, but at somewhat lower strength levels than hot-cold-worked material.

SESSION IV--September 19, 1956, at 1:45 p.m.

MOLYBDENUM ALLOYS FOR GAS TURBINE APPLICATIONS

Chairman: N. E. Promisel
Bureau of Aeronautics
Department of the Navy

DESIGN CONSIDERATIONS AND ENGINE TESTING OF MOLYBDENUM FOR AVIATION GAS TURBINES

R. T. Begley
Aircraft Gas Turbine Division
Westinghouse Electric Corporation

Molybdenum-base alloys are attractive to the jet engine designer primarily because of their outstandingly high temperature-strength properties. In order to establish more clearly the relationship of molybdenum-base alloys to future jet engine design, the effect of increased turbine-inlet temperatures on the performance of two types of aircraft gas turbines, the turbo-jet and the turboprop, is discussed.

The two basic methods of increasing turbine-inlet temperatures in aircraft gas turbines are through turbine cooling and the use of improved refractory materials. A number of problems are associated with both of these approaches, and depending on the type of engine and the application, either or both of these methods may be desirable.

At present, molybdenum-base alloys appear to hold the greatest potential for very-high-temperature jet-engine applications, despite the many serious problems still to be completely overcome. In addition to the well-known oxidation problem inherent in molybdenum and molybdenum-base alloys, the high thermal conductivity, relatively high density, and low coefficient of thermal expansion of molybdenum also present difficulties to the designer. The application of molybdenum to jet-engine design must be considered in relation to the effect of its physical properties on adjacent components. For example, the high thermal conductivity of a molybdenum turbine blade may cause a very considerable rise in the rim temperature of the turbine disc.

At present, effort is being concentrated on obtaining engine operating experience with molybdenum components. A great deal of work remains to be done in improving reliability, developing coating inspection techniques, and in producing high-strength joints. Major activity must be concentrated on developing molybdenum alloys and coatings which can operate successfully at temperatures considerably in excess of 2000°F.

THE PROTECTION OF MOLYBDENUM AGAINST HIGH-TEMPERATURE OXIDATION

J. J. Harwood
Metallurgy Branch
Office of Naval Research

The major obstacle limiting the use of high-strength molybdenum base alloys for many high-temperature military applications is their complete lack of resistance to oxidizing atmospheres at high temperatures. This paper reviews the status of the problem of the oxidation of molybdenum, and attempts to summarize the research programs which have been underway for the development of protective coatings. The requirements for protective coatings, with particular emphasis on gas-turbine applications, are presented, and the significant methods of testing coatings are described. The variety of protective coatings which have been investigated include metal cladding, and ceramic, sprayed, hot-dipped, vapor-deposited, diffusion and electroplated coatings and combinations thereof. The results of work on oxidation protection by alloying and the use of stable molybdates are discussed.

While a completely satisfactory coating has not yet been developed, recent work indicates that several types of sprayed coatings and electrodeposited multilayer coatings exhibit considerable promise for success within the temperature range of 1800-2000°F.

FORGING OF MOLYBDENUM-ALLOY TURBINE BUCKETS

J. J. Russ and G. E. Schrader
Steel Improvement & Forge Company

The increased need for higher strengths at higher and higher operating temperatures in present and advanced turbo-jet engine designs has brought about a joint development program by Climax Molybdenum Co., Westinghouse Electric Company (Aviation Gas Turbine Division), and Steel Improvement & Forge Company, to forge and engine-test molybdenum 0.5 percent titanium alloy turbine buckets.

This paper deals specifically with the material supplied and the forging procedures employed in the production of turbine bucket forgings.

While a number of problems remain to be solved, this program indicated that precision forgings to bucket configuration could be made in production with no major difficulties nor exorbitant manufacturing costs.

INVESTIGATION OF THE EFFECTS OF HOT-COLD WORK ON THE PROPERTIES OF MOLYBDENUM ALLOYS

J. R. Giancola and I. Perlmutter
Materials Laboratory
Wright Air Development Center

and

M. Semchyshen and R. Q. Barr
Climax Molybdenum Company

Two alloys, molybdenum-0.28 percent niobium and molybdenum-0.5 percent titanium, were investigated for the effect of fabrication variables on the degree of strain hardening and the resultant mechanical properties, and on subsequent resistance to softening with exposure to elevated temperatures. Fabrication was accomplished by either rolling or forging. The variables studied in the phase of the program dealing with rolling were composition, rolling temperatures, and amount of reduction. The variables encompassed by the forging studies were composition, rolling temperature of the bars from which specimens were taken, structural condition prior to forging, forging temperature, and amount of reduction.

The hardness of the rolled bars increased rapidly with increasing amounts of reduction by rolling, up to reductions of about 10 percent, then increased at a lower rate at successively higher reductions. When rolled bars were subjected to one-hour exposures at elevated temperatures, those sustaining the highest reductions displayed rapid declines in hardness and complete recrystallization in the range from 2250° to 2400° F for the niobium alloy and 2600° to 2800° F for the titanium alloy. Bars sustaining less reduction by rolling generally displayed a gradual approach to minimum hardness with increasing exposure temperature. At very low amounts of reduction, higher temperatures were required to remove final vestiges of strain from the niobium than from the titanium alloy. When severely strain-hardened bars were partially recrystallized, somewhat higher tensile strength and ductility values were recorded than when bars of the same alloy were rolled to develop the same hardness.

Forging studies revealed a rather complex dependence of degree of strain hardening upon composition, specimen condition prior to forging, forging temperature, and amount of reduction.

Specimens in the strain-hardened condition prior to forging developed substantially higher hardness than specimens partially or fully recrystallized prior to forging. At the highest forging temperature, 2400° F, specimens from the niobium alloy were less severely strain hardened than comparable specimens from the titanium alloy, owing to the relatively greater structural stability of the latter. A relationship was evolved between as-forged hardness and softening on exposure to elevated temperatures. Although substantial scatter was observed, the data are considered useful design criteria. The effects of time and temperature were found related by the expression

$$T(22.0 + \log t) = \text{constant},$$

where T is absolute temperature and t is time.

When specimens containing gradients in the degree of strain hardening were tested under tension over short periods of time at room temperature and 1800° F, they invariably failed at locations corresponding to minimum strain hardening. Certain creep-rupture tests at 1800° F, however, failed at locations corresponding to maximum strain hardening. Examination of these

specimens indicated that partial recrystallization and softening had occurred under the combined influences of time, temperature, and stress, at locations where no structural changes had taken place after exposures under similar conditions of time and temperature only.

Throughout the investigation, a consistent tendency was observed in which the higher the working temperature, the higher was the hardness, in direct contradiction to classic concepts. This apparently anomalous behavior was attributed to strain aging.

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